

SEMI-ANNUAL PROGRESS REPORT

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During the report period two research projects were phased out. They are Dr. S.C. Wait's project entitled, "Vibrational Spectroscopy of Inorganic Substances in the Vapor Phase," and Dr. R.A. Bailey's project entitled, "Thermal Decomposition of Inorganic Coordination Compounds." These studies will be continued at the Materials Research Center, but will be supported from other funds.

On April 23, 1966 the Materials Research Center was formally dedicated as part of a "Man and Materials Symposium" that was held on campus. The space and facilities provided by the new structure have enhanced Rensselaer's ability to conduct meaningful research and to educate graduate students.

RESEARCH PROJECTS

Mechanical Properties of Polymers 470.05

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A. Fracture of Polymers

Fracture studies on polymethylmethacrylate under conditions of compound, cyclical loading are being continued. Crack propagation and fracture morphology studies are now in progress and indicate several, perhaps three, distinct regions of crack velocity during fatigue failure of a glassy plastic. Additional data on the effects of frequency and load amplitude are being taken. A fracture theory for glass-like high polymers which accounts for a time and path dependent stress concentration factor arising from localized viscoelastic-plastic deformations was reported previously. Three manuscripts have resulted from this study and will be submitted for publication shortly.

Theoretical studies on the interactions of inclusions in glass-like matrices are being undertaken. In order to characterize the mechanical properties of a glass-like matrix-rubber dispersed phase composite system, it is necessary to obtain the stress concentrations and dilatational fields in the vicinity of the dispersed phase particles. In particular, the interactions of the stress and dilatational fields, when the glassy matrix is undergoing localized ductile deformation between the inclusions, are of primary importance to the fracture and impact strength of these materials. Experimental studies on the mechanical properties of composite systems are being initiated under a grant from the National Institute of Dental Research.

Experimental studies on the effect of a void on the stress crazing ability of glass-like plastics have shown that a decrease of dilatational field, as a result of the void, results in a decrease of craze formation. From the observed areas of no craze formation and the applied sample stress, calculations of the minimum required dilatation for craze initiation have been made. The values obtained agree, within experimental error, with the dilatation required to exceed locally the glass transition.

B. Viscoelastic Properties of Polymers

Analysis of the swelling and absorption isotherms for polystyrene, polymethylmethacrylate, and polycarbonate has indicated a need for thermodynamic interaction parameters of polymer-solvent systems at constant volume. Accordingly, a pressure cell has been designed to subject solid polymers to various solvents and to maintain the solid at constant volume. The data consist of liquid volume absorbed and solid "pressure" required for constant volume, both as functions of time. The apparatus will be tested shortly.

Dielectric spectra on the copolymer system poly (vinyl alcohol-acetate) of compositions ranging from 0 to 100% acetate, and over a temperature range of -190° to 100° C and frequency range 50cps to 600,000 cps have been obtained. Multiple transitions, including α , β and γ types, have been observed. Also, transitions in the activation energy of the β transition have been found. The data indicate large scale motions can occur while the sample is well below the glass-transition temperature. Further studies are in progress. The mechanical spectra studies of these polymers are in progress, as are the high pressure x-ray studies of several polymers.

A Study of the Interaction of Dislocation with the Discrete Second-Phase Particles in Dispersion-Strengthened Alloys

470.09

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The objective of this program has been to delineate the effects associated with the presence of a distributed second-phase in a crystalline matrix by both theoretical considerations and experimental observations.

During this period, research efforts have centered principally upon experimental observations utilizing both replication and thin film transmission electron microscopy techniques. The specific areas investigated include:

A. Recovery and Recrystallization Kinetics

Recent work in this laboratory has demonstrated that the recovery and recrystallization behavior of two-phase alloys is composed of two parts. In particular, the thermal effect on the cold worked structures can be described as follows: At approximately 0.5 of the absolute melting temperature, localized shifting of the individual dislocations within cell walls produce changes in relative cell-to-cell misorientation and dislocation density. At higher temperatures, cell wall migration results in wall disintegration, hence cell growth occurs. In order to determine the generality of this recovery and recrystallization mechanism, studies of recrystallization behavior in high purity aluminum have started, utilizing hot stage transmission electron microscopy and cine' sequence recording techniques. To date, most effort has been directed toward the definition of standard substructural configurations produced by cold work. This is particularly important in order to be able to delineate the progress of dislocation rearrangement during the recovery process. This study is currently in progress.

B. Fracture Behavior

The influence of a distributed phase upon fracture mode and crack propagation is being studied in steel. Replication studies of the fracture surfaces of these alloys broken in fatigue are being used to establish the mechanism by which dissolved gasses, particularly hydrogen, affect fatigue fracture. This program is currently in progress and is being handled in cooperation with Dr. H. Rogers of the General Electric Research Laboratory.

C. Precipitation Kinetics

The formation of quench and solute-induced defect structures apparently controls the nucleation and growth kinetics of many precipitate phases. This present study is concerned with the nature of such heterogeneous nucleation kinetics upon precipitation using Al-Ag and Al-Ag-4 alloys as model alloy systems. Currently, this area is being pursued with a dual emphasis on both the formation of the defect structure and the influence of this defect structure upon the nucleation and growth mechanisms involved in the formation of the γ' phase. Transmission electron microscopy has

shown that several types of defect structures can be generated in these alloy systems, each of which can act in the formation of the γ' phase. While the final phase morphology is rather insensitive to the origin of phase formation, the initial γ' nucleation is uniquely defined by the original defect structure. This study is currently in progress.

An apparatus has been constructed which provides for austenitizing steel specimens, quenching to just above the M_s temperature, performing tensile tests at this temperature, and then quenching to form martensite. During the final quenching, the progress of the transformation is followed by continuous magnetic response measurements on the specimen. Thus, the effect of the flow stress of the prior austenite, as modified by work hardening, on the kinetics of the martensite transformation can be determined. This investigation has shown that the M_s temperature is a direct function of the flow stress. This work is now being extended in order to further investigate the transformation kinetics.

Previously, in this laboratory, it was shown that very fast rates of quenching from austenite increased the temperature for the start of the martensite transformation. This work was interpreted as reflecting the degree to which carbon segregated during the quenching process and thereby influenced the shear strength of the austenite. This study now has been extended to Fe-C-x alloys, in order to determine the effects of third elements upon this behavior.

D. Martensite Transformation

The kinetics of the martensite transformation in the Fe-C system have proved elusive. As a result of recent work in this laboratory, it is felt that this transformation may be controlled by the shear strength of austenite. For this reason an investigation of the kinetics of the martensite transformation as a function of austenite strength has been undertaken.

E. Dispersion-Strengthening

Two studies are being undertaken in order to further elucidate the mechanism by which a dispersed second phase enhances the mechanical properties of crystalline solids.

The first is a study of the influence of particle size, shape, and interface upon the yield strength of these alloys. A nickel base alloy containing the Ni_3Al phase has been chosen as a model alloy system for this study. By changing the heat treatment schedule, the shape, size and interface characteristics of the dispersed phase can be altered. Currently, transmission electron microscopy and replication techniques are being used to quantitatively determine the dispersed phase morphology. After this phase of the program, the mechanical behavior of similarly processed specimens will be determined and the results correlated with the structural data.

The second program deals with a basic hypothesis of the strengthening models which have been proposed to account for the effect of a distributed phase. Is second phase particle shear a requirement for the yielding behavior of an alloy containing a fine distribution of hard particles? A transmission electron microscope investigation in this area has been initiated.

F. Theoretical Studies

Theoretical studies of the strengthening mechanisms in solids based upon dislocation theory are progressing on a continuous basis.

Mechanisms of Solidification

470.10

Senior Investigator:	W.J. Childs, Ph.D. Professor of Metallurgical Engineering
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A general review of the nucleation effects of mechanical vibration on supercooled bismuth has been completed. It has been demonstrated that the nucleation occurs over a wide frequency range only when cavitation has occurred. Factors affecting the degree of cavitation have been explained.

At 20,000 cycles/second the vibrational unit was designed to give accoustical cavitation. In all cases of accoustical cavitation nucleation was initiated at any values of supercooling greater than 10 C° for bismuth. A model to explain the nucleation during cavitation of metals such as bismuth that expand on freezing has been proposed on the basis of large negative pressures which occur following the compression wave.

Vibrations of very high frequency; e.g., 2.5 megacycles, where cavitation has not occurred have been shown to increase supercooling and therefore presumably deactivate some nucleating agents which may be present.

Ultrasonic Research

470.11

Senior Investigator:	H.B. Huntington, Ph.D. Professor of Physics
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The interaction of a longitudinal ultrasonic pulse at 15 Mcs with a tranverse pulse at 10 Mcs to produce a longitudinal pulse at 25 Mcs, and the interaction of two transverse ultrasonic pulses at 10 Mcs to produce a longitudinal pulse at 20 Mcs, have been observed in aluminum. A fused quartz disc and discs of single crystal sodium chloride have been procured for further investigations.

The plan for the beam-interaction work is to observe the interaction for a series of frequency ratios in the isotropic specimens (aluminum and fused quartz) attempting to get a semiquantitative measurement of the third-order elastic constants. This will then be carried over to the single crystal sodium chloride specimens.

Several of the velocities necessary for determining the second-order elastic constants of anthracene have been measured but the data are only preliminary.

Ultrasonic Pulse Interferometry

470.12

Senior Investigator: S. Katz, Ph.D.
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During the past six months the study of the properties of metal halides, especially AgI, in a large pressure gradient has continued. The results have been described in detail in one doctoral dissertation (R.N. Schock) and are being prepared for publication. In a high-pressure cell utilizing diamond anvils these substances are observed to dissociate, with the metal ions migrating toward the low-pressure area and the halide ions toward the high-pressure area. This effect is attributed to the large difference in compressibility between the two constituents and to the small activation energy for grain-boundary diffusion. Experiments involving the spiking of AgI with I_2 and the annealing of AgI prior to application of pressure confirm these interpretations.

Ultrasonic studies of materials with polymorphic transitions (Bi, Ni, KCl, RbCl) have demonstrated the necessity of monitoring piston displacements in order to obtain meaningful information on density and elastic constants for these materials. The modifications required to carry out this measurement have been undertaken. Reliable calibrations have been obtained to 90 kb of the high-pressure apparatus, utilizing electrical transitions in Ni, Bi, Tl, and Ba.

The Relation of Molecular Structure and Intermolecular 470.15
Action in Flow of Polymers and Polymer Dispersions
as a Function of Temperature and Shear Rate

Senior Investigator: W.H. Bauer, Ph.D.
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Research Staff: Theodore Tsakumis, Ph.D.
Ford Fellow

The objective of the research has been to relate the flow properties of liquid polymers related to polybutadienes with the molecular structure and other properties of the polymers.

A series of poly-dimethyl-siloxanes of varying molecular weight was studied with the use of a cone plate viscometer. Instrumentation was developed for measurement of special properties.

It is planned to examine the flow properties of solutions of polybutadienes in appropriate solvents with the expectation that the flow properties will be related to the molecular structure.

The Influence of Deformation Mode on the Ductile 470.18
to Brittle Transition of FeCo-V Alloys

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Allegheny Ludlum Research Fellow

Kenneth Jordan, Research Assistant, has been studying the role of changes in deformation modes (slip and twinning) on the ductile to brittle transition of an FeCo-2%V superlattice alloy. The effect of temperature, from -196°C to 25°C , on yield stress, fracture stress and ductility is much stronger for disordered than for ordered material. Preliminary results from transmission electron microscopy have shown a difference in the dislocation structure with ordering: disordered samples show cell structure, while ordered samples exhibit a uniform distribution of dislocations. Additional work is to be performed in an effort to relate this observation to the ductility and fracture mode, and to extend the test results to 500°C .

Andrew Blackwood, Allegheny Ludlum Research Fellow, has initiated a project to determine the mechanism of stress corrosion cracking in copper-base alloys. Several copper-aluminum alloys have been prepared by vacuum melting. These have been cold-rolled to 0.030 in. strip, from which stress corrosion specimens are to be made. A creep stand has been modified for use as a stress corrosion rig. The objective of the test program to be carried out in the next report period is to determine the relative importance of surface oxide films and of the dislocation substructure produced by deformation on the incidence of stress corrosion cracking in these alloys.

Low Temperature Physics 470.19

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G.I. Kaufman and J.S. Willis (supported by the U.S. Army) have received Ph.D. degrees for work partly supported by this project during the past six months. Their thesis topics were, respectively, "Annealing of Liquid Nitrogen Temperature Plastic Deformation and Radiation Damage in Lead" and Resistive Transitions and Critical Current Curves of Type II and Type I

Superconductors." The former is an attempt to understand the basic lattice defects in lead and has employed the techniques of stored energy release, creep, and ultrasonic attenuation. This last technique has been analyzed to yield the motional energies of the various defects active between 77° K and 300° K, with the result that the binding energy of interstitial atoms to impurity atoms appears to be 0.15 ± 0.01 eV, and the motional energy of vacancies in lead is 0.31 ± 0.04 eV. Further work is continuing in applying the technique to other materials and in understanding lead more completely.

The superconducting study involved the introduction of lattice defects into samples by very low temperature deformation and observation of variations in the current carrying properties. A theory involving the resulting inhomogeneous electron scattering in the samples has been developed. This approach appears to be capable of yielding most of the range of superconducting characteristics from a small number of parameters. Further development is planned.

The ultrasonic attenuation studies of the superconducting energy gaps of Pb, Hg, and Tl mentioned in earlier reports are nearing completion. Interesting frequency dependent effects have been observed in the first two materials which are now being studied as a function of small impurity additions and γ -irradiation.

Pyrolytic Materials 470.20

Senior Investigator:	R.J. Diefendorf, Ph.D. Associate Professor of Metallurgical Engineering
Research Staff:	B. Butler, B.S. Graduate Assistant A. Kinawi, M.Sc. Graduate Assistant

The deposition of pyrolytic graphite from methane gas has been found to be enhanced by small additions of boron trichloride. Although the magnitude of this catalysis is known to vary with deposition conditions, experiments performed under apparently identical conditions have often given different results. A possible explanation is the presence of varying amounts of impurities in the feed stock gases. Because of the value of boron trichloride in altering deposition profiles and in improving mechanical properties, a research program was initiated to determine the effect of impurities on the boron trichloride catalyzed deposition of pyrolytic graphite.

The deposition of pyrolytic graphite is a complicated process which, for the particular case, has fourteen variables. For a three-level fourteen-dimension matrix set of experiments, approximately 100 million experiments would be necessary to completely determine all interactions. The problem was eased by just studying the effects of impurities in natural gas (CO_2 , N_2 , C_2H_6 , O_2 , C_3H_8) on the deposition. Even with this simplification, a large number of experiments must be performed and analyzed. A new rapid technique was evolved to evaluate interactions, and a simple way to handle the data was found.

The results were that the higher hydrocarbons (C_2H_6 , C_3H_8) found in natural gas were as effective catalysts in pyrolytic graphite deposition as was boron trichloride. We plan to study the mechanisms for these catalyses in the near future. A mass spectrometer which was donated to us should be helpful.

Dispersion-Strengthened Materials 470.23

Senior Investigator: F.V. Lenel, Ph.D.
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A paper entitled "Observations on the Hardness, Lattice Parameter and Microstructure of Internally Oxidized Silver Magnesium and Silver Aluminum Alloys," by J.S. Hirschhorn and F.V. Lenel, based on Dr. Hirschhorn's work on this project, was published in the June 1966 issue of ASM Transactions Quarterly. A second paper entitled "The High Temperature Steady State Creep of Pure Silver and Internally Oxidized Silver Magnesium Alloys," by G.R. Leverant, F.V. Lenel, and G.S. Ansell, based on Dr. Leverant's work on this project, has been submitted to ASM Transactions Quarterly for publication.

Dr. H.S. Nayar's thesis, entitled "Creep behavior of Pure Ice and Ice Dispersed with Ultrafine Amorphous Silica (SiO_2) Particles," was accepted for the Ph.D. degree in June 1966. This work was part of the project on dispersion strengthening. Dr. Nayar's results may be summarized as follows:

A technique was developed to produce ice containing a fine and uniform distribution of amorphous silica particles. The structure of the ice alloy was stable up to a temperature just below the melting point of ice. The temperature and stress dependence of the minimum creep rate was determined for ice containing silica particles. The minimum creep rates were measured in the temperature range of -22 to $-2^\circ C$, and in the stress range of 4.64 to 17.8 bars (1 bar = 1.0×10^6 dyne/cm² \approx 14 psi) under tension for samples with 1 volume percent of silica.

Within these ranges of temperature and stress, it was found that ice containing 1 volume percent of dispersed silica particles creeps 10-40 times slower than pure ice.

On the basis of the observed temperature and stress dependence of the creep rate, it is proposed that the strengthening mechanism in ice dispersed with 1 volume percent silica particles is the cross-slip of dislocations over the dispersed particles.

The work on the properties of lead strengthened by a fine dispersion of lead oxide was completed. Creep, tensile, overcharge, and potentiostatic anodic dissolution behavior was obtained for a standard lead-acid battery

grid material (7% Sb-. 6% Sn-. 6% As-Pb), one or more dispersion-strengthened materials and, in some cases, pure lead. A comparative analysis of the results yielded the following conclusions: (1) the creep, tensile and overcharge properties of dispersion-strengthened lead are superior to those of cast 7%Sb-Pb; (2) dispersion-strengthened lead and pure lead do not exhibit similar electrochemical properties.

In June 1966 a conference on oxide dispersion-strengthening organized by the Hudson-Mohawk Section and the High Temperature Alloys Committee of the Metallurgical Society of AIME was held at Bolton Landing, New York, for which F.V. Lenel was program chairman. Partially as a result of the presentations and discussions at this meeting, it was decided to plan a new phase of the dispersion-strengthened material project for the coming academic year, in which the properties of cold-worked and recrystallized dispersion-strengthened systems will be studied.

Metallic Dissolution 470.25

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The purpose of this program is to determine the basic corrosion and electrochemical characteristics of iron and iron alloys (steels).

Current research includes anodic and cathodic polarization experiments on a number of alloys. Effects of the phosphorus-, carbon-, and manganese-contents on electrochemical parameters have been determined. Analysis of these data is in progress.

Nuclear Magnetic Resonance Research 470.26

Senior Investigator: P.A. Casabella, Ph.D.
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When an ion is placed in an electric field gradient the electrons in the ion polarize and result in a larger field gradient at the nucleus than would exist without the electrons. In order to analyze experimental results the amplification of "antishielding" factor of the ion must be known. Experimental and theoretical values of this factor were in poor agreement for many ions in solids when this study was begun.

In this work a known electric field gradient was produced by straining a crystal and studying a nucleus at a site of zero field gradient in the unstrained crystal. By measuring the field gradient at the nucleus it is possible to determine the antishielding factor. Such studies have been completed for all ions in NaCl and NaBr. By taking into account the effects of overlap between ions in the solid, good agreement between theory and these experiments has been obtained.

X-Ray Scattering

470,28

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The principal effort in the present program has been directed toward the investigation of the lattice dynamics of metals and alloys by x-ray diffraction techniques. Measurements of the thermal variation of the x-ray Debye temperature of single crystals of pure nickel and chromium in the temperature range $100^{\circ}\text{K} < T < 520^{\circ}\text{K}$ have been completed; a paper reporting on this work is in press in Acta Crystallographica. A similar study on single crystals of zinc has been carried out from 520°K to 40°K ; this work presently is being extended down to approximately 3.5°K using our liquid helium, x-ray cryostat.

Improved calculations of the thermal diffuse x-ray scattering corrections are almost completed. Experimental studies of such diffuse scattering are being planned to determine elastic behavior as well as to verify both these new and the previous approximate calculations.

In addition, theoretical lattice dynamical studies in conjunction with our experimental work are in progress. These studies primarily involve interatomic force model calculations (Begbie-Born model, chiefly) of the x-ray Debye temperature, heat capacity, and dispersion curves for Ni, Fe-33%Ni and Cr. For nickel, these calculations represent the experimental heat capacity, x-ray Debye temperature, and dispersion curves, to within 10%.

Studies of the crystal and molecular structure of the coordination compounds of the platinum group metals are in progress.

The real time computer control of an automated full circle diffractometer is near completion. This will be tested shortly for use in both the thermal diffuse scattering and single crystal structure studies.

Thermal Properties of Polymers

470.29

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A. Thermal Conductivity

The results of thermal conductivity measurements on oriented polymers show the conductivity to increase with orientation but to a lesser degree than predicted earlier from calculations based on theory developed by Hansen and Ho. To resolve this discrepancy, new calculations will be made using more exact descriptions of molecular geometry in the oriented polymer.

B. Heat Transfer in Porous Media

The first part of this extensive study on simultaneous heat and mass transfer in porous media has been completed. A set of three papers is being prepared for publications, the first is a review, the second presents a new theoretical analysis of the subject, and the third compares new experimental observations with this analysis. The experimental work to date has been confined to water-air systems below 120°C. Further experimental work with other materials and at higher temperatures is planned. (Supported by the National Science Foundation).

C. Crystallization

Studies of the effects of crystallization conditions on polymer morphology as observed by electron microscopy of thin sections is continuing. Polyethylene-terephthalate has been crystallized under a variety of temperature conditions while monitoring the crystallization rates. Evaluation and comparison of the structure of these specimens is in progress.

D. Cold Drawing

Electron microscopy of thin sections of Nylon 66 before and after cold-drawing show the micro fibrils (crystals) to have decreased in thickness from 100 Å to 30 Å as a result of cold-drawing. This direct observation of changes in crystal thickness supports the postulate of intra-crystalline slip as primary deformation mode in elongation and orientation of partially crystalline polymers. This work is continuing to seek more detailed characterization of morphological changes on cold-drawing. (Supported by the Army Research Office-Durham).

Glass and Non-Metallic Materials 470.30

Senior Investigator: J.D. Mackenzie, Ph.D.
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The objectives of this work are twofold: (a) to correlate structures and properties of glasses and other non-metallic solids, and (b) to prepare new non-metallic materials and study their properties.

During this period, research has been carried out on (1) semiconducting glasses, (2) wetting of liquid metal on ceramics, (3) strength of composites, and (4) vapor deposition of nitrides. These projects are still in progress and will be continued.

Vibrational Spectroscopy of Inorganic Substances 470.31
in the Vapor Phase

Senior Investigator: S.C. Wait, Jr., Ph.D.
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Research Staff: G. Kelly, B.A.
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The measurements of the infrared spectra of molten inorganic nitrates are being continued. In addition to the pure nitrates, mixtures of AgNO_3 with AgCl and AgBr are being studied to examine the effect of varying anionic fields on the nitrate ion spectrum. In these mixtures the symmetric stretching frequency of the nitrate ion is enhanced.

Studies directed toward establishing a satisfactory potential function for molten salts are being undertaken. Preliminary results show that a multiple-well potential surface is generated as the interionic distance increases. The possibility of explaining observed frequencies not due to the nitrate ion is being considered from the standpoint of such anion-cation interactions.

Irreversible Thermodynamics of the Solid 470.32
State of Linear High Polymers

Senior Investigator: B. Wunderlich, Ph.D.
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Research Staff: F. Hamada, Ph.D.
Postdoctoral Research Assistant
S. Wolpert
Graduate Assistant

The measurements of the time dependent specific heats of glasses in the transition region are being continued after an interruption during the three summer months.

A new hot-stage with long term stability of $+ 0.01^{\circ}\text{C}$ for the range 0 to 150°C has been built and tried out. It will be used to study size dependent melting of paraffin and polymer single crystals during the next three months.

The melting behavior of crystals of polyoxymethylene is being investigated using the DTA apparatus purchased under the above account. It is expected this work will be completed by December 1966. Preliminary results show that perfect polyoxymethylene single crystals can be superheated easily, similar to polyethylene and polytetrafluoroethylene (see September 1965 report). The irreversible reorganization of less perfect crystals is also studied.

Ultra-Low Temperature Solid State Physics Research 470.33

Senior Investigator: G.L. Salinger, Ph.D.
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The object of this program is to measure the thermal properties of magnetic salts below 4°K . In particular, thermal conductivity will be measured in order to determine interactions in the magnetic and lattice systems.

During the past six months the cryostat has been tested. The lowest temperature reached was 0.4°K with the He^3 refrigerator. The systems to measure temperature by means of paramagnetic salts and radio resistors also have been tested.

The thermal conductivity apparatus is being planned. Suitable hydrated crystals are being grown.

Thermal Decomposition of Inorganic 470.34
Coordination Compounds

Senior Investigator: R.A. Bailey, Ph.D.
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Research Staff: I.R. Feins, B.S.
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The objectives of this research were to study the products of thermal decomposition of coordination complexes containing organic ligands in order to establish the patterns of thermal break-up, and the factors controlling thermal stability, of this class of compound. The compounds initially chosen,

those of substituted ureas with first-row transition and post-transition metals, proved to be of interest in other ways; their structures and significant new data have been obtained.

Decomposition studies have been carried out for a series of these complexes - chiefly the compounds of methylurea, sym-dimethylurea, asy-dimethylurea, and phenyl-urea with the chloride of Co, Ni, Cu, Zn, Cd, and Hg. Correlation of the data obtained, and identification of some of the decomposition products, are still under way.

Thermodynamics and Kinetics of Vaporization 470.35
Processes of Inorganic Materials

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The objectives of this work are to determine thermodynamic and kinetic properties of vaporization processes of transition metal chalcogenides. Experimental procedures include knudsen- and langmuir-type studies on poly- and single-crystalline materials.

Single crystals of manganese sulfide and selenide have been prepared using chemical transport techniques. Synthesis of manganese telluride crystals is in progress.

A vacuum-thermo-balance has been set up, and test runs proved to be successful. This system now is being used for equilibrium studies on MnSe.

Solid solution in the ternary Mn-Cd-S system is presently being investigated over the entire range of composition and at various temperatures. These studies are carried out on poly-crystalline samples employing x-ray diffraction techniques.

The equilibrium studies will be extended to an investigation of the mechanism of vaporization.

Selected composition in the solid solution range will be used to study the effect of composition on single crystal growth.

Electron-Spin Resonance 470.37

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Graduate Assistant

Further emphasis has been placed upon the development of a highly sensitive, slow wave, helical sample structure for double resonance (ENDOR) and optical studies. Various modes of helix operations have been analyzed, and a system with characteristics approaching ideal performance has been achieved.

In order to examine activation and annealing processes in materials over a wide temperature range a variable temperature unit, compatible with both NMR and ESR spectrometers, has been developed. This system, using nitrogen as a heat exchange gas, is capable of maintaining controlled temperatures over the range from 80°K to 500°K.

Preliminary measurements have been carried out at room temperature on the V^{4+} paramagnetic properties in several glass systems. With fairly high concentrations on V^{4+} a very broad, unstructured, resonance spectrum is obtained. Further studies will be conducted at elevated temperatures in order to determine the origin of this broadening which, it is suspected, is due to exchange effects. At lower concentrations of V^{4+} a narrower, fairly complex spectrum is obtained which may be characteristic of the glass matrix. Further analysis and measurements are being carried out in order to determine the environment of the V^{4+} ions.

Polymer Characterization 470.38

Senior Investigator: F.W. Billmeyer, Jr., Ph.D.
Professor of Analytical Chemistry

Research Staff: N.K. Blackwood, B.S.
Graduate Assistant
E.C. Rhodes
Graduate Assistant

A photometer designed for the precise calibration of photometric scales by the method of addition of light fluxes was constructed, tested, and used to calibrate the photometric scale of a General Electric spectrophotometer.

Future work on this project will be directed toward the synthesis and characterization of polymers; work on color measurements is being supported from other sources.

TECHNICAL PAPERS SUBMITTED FOR PUBLICATION

Casabella, P.A., Marsh, J.L., Jr.

"The Gradient-Elastic Tensor in Sodium Chloride and Sodium Bromide"
Submitted to Physical Review

Katz, Samuel

"High-Pressure Research and Geophysics, A Status Report"
Published in the Transactions, American Geophysical Union

Wunderlich, B., Prime, Bruce

"The Equilibrium Melting of Polymers"
Will be presented at the International Union of Pure & Applied
Chemistry and published in the proceeding of the conference
(Journal of Polymer Science)

Shaw, R.W., Schenck, J.F., Willis, J.S.

"Resistance in the Mixed State of Type II Superconductors"
Submitted to Physical Review

Shaw, R.W., Kaufman, G.I., Woo, D.S.

"The Annealing of Liquid Nitrogen Temperature Plastic Deformation
and Radiation Damage in Lead"
Submitted to Acta Metallurgica

Wunderlich, B., Cormier, C.M.

"The Heat of Fusion of Polyethylene"
Submitted to Journal of Physical Chemistry

Raman, Sri

"Patterson Functions and Vector Sets"
Published in the Transactions of the American Crystallographic
Association

APPENDIX A

Members of Interdisciplinary Materials Research Center
Faculty Committee

S.E. Wiberley, Chairman	Professor of Analytical Chemistry and Dean of the Graduate School
G.S. Ansell	Professor of Metallurgical Engineering
W.H. Bauer	Professor of Physical Chemistry and Dean of the School of Science
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G.J. Janz	Professor of Physical Chemistry, Chairman of the Department of Chemistry
F.V. Lenel	Professor of Metallurgical Engineering, Chairman of the Department of Materials
S.S. Sternstein	Associate Professor of Chemical Engineering
J.M. LoGiudice	Administrative Director of the Interdisciplinary Materials Research Center